SECTION 9
WASTEWATER SAMPLING

SECTION OBJECTIVE:

- To provide guidance for the proper collection of wastewater samples.

9.1 Introduction

The variety of conditions at different sampling locations require that considerable judgment be exercised regarding the methodologies and procedures for the collection of representative samples of wastewater. Each sampling location warrants attention commensurate with its complexity. There are, however, basic rules and precautions generally applicable to sample collection. Acceptable procedures are generally those outlined in the NPDES Compliance Inspection Manual (1). Some important considerations for obtaining a representative wastewater sample include:

- The sample should be collected where the wastewater is well mixed. Therefore, the sample should be collected near the center of the flow channel, at approximately 40 to 60 percent of the water depth, where the turbulence is at a maximum and the possibility of solids settling is minimized. Skimming the water surface or dragging the channel bottom should be avoided. However, allowances should be made for fluctuations in water depth due to flow variations.

- In sampling from wide conduits, cross-sectional sampling should be considered. Rhodamine WT dye (See Section 15.7 for procedures) may be used as an aid in determining the most representative sampling locations.

- If manual compositing is employed, the individual sample portions must be thoroughly mixed before pouring the individual aliquots into the composite container. For manual composite sampling, the individual sample aliquots should be preserved at the time of sample collection (2).

- When collecting samples or installing sampling equipment, field investigators should always wear a new pair of the appropriate protective gloves (disposable latex gloves, rubber gloves, etc.) to prevent contamination of the sample and reduce exposure to hazardous substances.

9.2 Site Selection

Where applicable, wastewater samples should be collected at the location specified in the NPDES permit (if the source has such a permit). In some instances the sampling location specified in the permit, or the location chosen by the permittee, may not be acceptable for the collection of a representative wastewater sample. In such instances, the investigator is not limited by permit specifications and may collect a sample at a more representative location. When a conflict exists between the permittee and the regulatory agency regarding the most representative sampling location, both sites should be sampled, and the reason for the conflict should be noted in the inspection or study report and field notes. Recommendations and reasons for a change in sampling locations should be given to the appropriate permitting authority.

9.2.1 Influent

Influent wastewaters are preferably sampled at locations of highly turbulent flow in order to ensure good mixing; however, in many instances the most desirable location is not accessible. Preferable influent wastewater sampling locations include: 1) the upflow siphon following a comminutor (in absence of grit chamber); 2) the upflow distribution box following pumping from main plant wet well; 3) aerated grit chamber; 4) flume throat; 5) pump wet well when the pump is operating; or 6) downstream of preliminary screening. When possible, influent samples should be collected upstream from sidestream returns.

9.2.2 Effluent

Effluent samples should be collected at the site specified in the permit, or if no site is specified in the permit, at the most representative site downstream from all entering wastewater streams prior to discharge into the receiving waters. If a conflict exists between the permittee and inspector regarding the source being sampled or the location of the most representative site, follow the procedures previously described under “Site Selection”.

9.2.3 Pond and Lagoon Sampling

Generally, composite effluent wastewater samples should be collected from ponds and lagoons. Even if the ponds or lagoons have long retention times, composite sampling is necessary because of the tendency of ponds and lagoons to have flow paths that short circuit and changes the design detention time.

9.3 Sample Types

For NPDES sampling, two types of sampling techniques are used: grab and composite. For these procedures, the NPDES permit specifies the appropriate sample type. A complete description of all NPDES sampling procedures and techniques is presented in the NPDES Compliance Inspection Manual (1).

9.3.1 Grab Samples

Grab samples consist of either a single discreet sample or individual samples collected over a period of time not to exceed 15 minutes. The grab sample should be representative of the wastewater conditions at the time of sample collection. The sample volume depends on the type and number of analyses to be performed.

9.3.2 Composite Samples

Composite samples are collected over time, either by continuous sampling or by mixing discrete samples. A composite sample represents the average wastewater characteristics during the compositing period. Various methods for compositing are available and are based on either time or flow proportioning. The choice of a flow proportional or time composite sampling scheme depends on the permit requirements, variability of the wastewater flow or concentration of pollutants, equipment availability, and sampling location. The investigator must know each of these criteria before a sampling program can be initiated. If an investigator knows or suspects that there is significant variability in the wastewater flow or if the investigator knows nothing about the facility, a flow proportional sample is preferable. Otherwise, a time composite sample would be acceptable.

A time composite sample consists of equal volume discrete sample aliquots collected at constant time intervals into one container. A time composite sample can be collected either manually or with an automatic sampler.

A flow proportional composite sample can be collected using one of two methods. One method consists of collecting a constant sample volume at varying time intervals proportional to the wastewater flow. For the other method, the sample is collected by varying the volume of each individual aliquot proportional to the flow, while maintaining a constant time interval between the aliquots. Prior to collecting flow proportional samples, the facility's flow measuring system should be examined for proper installation and accuracy (see Section 18). If the facility's primary flow measuring device does not meet standard conditions (see Section 18), or is in an unsafe or inaccessible location, then the investigator may collect time composite samples. If the flow measurement system is acceptable, samples should be collected using the appropriate flow proportioning methods.

Flow proportional samples can be collected with an automatic sampler and a compatible flow measuring device, semi-automatically with a flow chart and an automatic sampler capable of collecting discrete samples, or manually.

9.4 Use of Automatic Samplers

9.4.1 Introduction

Automatic samplers may be used to collect composite or grab samples when several aliquots are to be collected at frequent intervals or when a continuous sample is required. For composite sampling applications, the automatic samplers may be used to collect time composite or flow proportional samples. In the flow proportional mode, the samplers are activated by a compatible flow meter. Flow proportional samples can also be collected using an automatic sampler equipped with multiple containers and manually compositing the individual sample portions proportional to the flow (1).

Automatic samplers must meet the following requirements:

- Sampling equipment must be properly cleaned to avoid cross-contamination which could result from prior use (see Appendix B for cleaning procedures).
- No plastic or metal parts of the sampler shall come in contact with the water or wastewater stream when parameters to be analyzed could be impacted by these materials.
- The automatic sampler must be capable of providing adequate refrigeration during the sampling period. This can be accomplished in the field by using ice.
- The automatic sampler must be able to collect a large enough sample for all parameter analyses.
- The individual sample aliquot must be at least 100 mls.
- The automatic sampler should be capable of providing a lift of at least 20 feet and the sampler should be adjustable since the volume is a function of the pumping head.
- The pumping velocity must be at least 2 ft/sec to transport solids and not allow solids to settle.
- The intake line leading to the pump must be purged before each sample is collected.
- The minimum inside diameter of the intake line should be 1/4 inch.
- An adequate power source should be available to operate the sampler for the time required to complete the project. Facility electrical outlets may be used if available.
- Facility automatic samplers should only be used if 1) field conditions do not allow for the installation of EPA sampling equipment, and 2) the facility sampling equipment meets all of the requirements of this SOP.

Specific operating instructions, capabilities, capacities, and other pertinent information for automatic samplers are included in the respective operating manuals.

9.4.2 Conventional Sampling (Inorganic Parameters)

Conventional sampling includes all inorganic parameters (e.g., BOD_5 , TSS, COD, nutrients, and metals) that can be collected using an automatic sampler.

New tubing (Silastic®, or equal, in the pump and either Teflon® or Tygon®, or equal, in the sample train) will be used for each sampler installation.

Installation procedures include cutting the proper length of tubing, positioning it in the wastewater stream, and sampler programming. Protective gloves should be worn to reduce exposure and to maintain the integrity of the sample.

For a time composite sample, the sampler should be programmed to collect at least 100-milliliter aliquots at a frequency that provides a representative sample and enough sample volume to conduct all required analyses.

For a flow proportional sample, the sampler should be programmed to collect a minimum of 100 milliliters for each sample aliquot with the interval predetermined based on the flow of the monitored stream.

At the end of the compositing period, the sample collected should be properly mixed and transferred into the respective containers, followed by immediate preservation, if required. For routine inspections, the permittee should be offered a split sample.

9.4.3 Metals

When an automatic sampler is used for collecting samples for metals analyses, the entire sampler collection system should be rinsed with organic/analyte free water, and an equipment blank should be collected. Approximately one gallon of rinse water should be pumped through the sample tubing into the composite container and discarded. Nitric acid must be added to the metals blank container for proper preservation. The sampler may then be positioned in the appropriate location and the sampler program initiated.

If the sampler tubing is attached to a metal conduit pipe, the sampler intake tubing should be carefully installed upstream and away from the conduit to prevent metals contamination. This can be accomplished by clamping the tubing upstream of the conduit using laboratory clamps and wrapping the submerged portion of conduit pipe with a protective barrier (e.g., duct tape).

9.4.4 Extractable Organic Compounds, Pesticides, and PCBs

When an automatic sampler is used for collecting samples for the analyses of extractable organic compounds, pesticides, and/or PCBs, the installation procedures include cutting the proper length of new Teflon® tubing, rinsing of the entire sampler collection system with organic/analyte free water, and collection of appropriate blanks for organic compounds analysis. For the organic/analyte free water rinse, approximately one-half gallon is initially pumped into the composite sample container and discarded. An additional one and one-half gallons are then pumped into the composite sample container for distribution into the appropriate blank container. Finally the collection tubing should be positioned in the wastewater stream and the sampler programmed and initiated.

9.4.5 Automatic Sampler Security

Field investigators should take whatever steps are necessary to prevent tampering with EPA equipment. A lock or custody seal may be placed on the sampler to detect tampering. However, this does not prevent tampering with the sample collection tubing. If necessary, seals may be placed on the sampling pole and tubing line to further reduce tampering possibilities.

9.4.6 Automatic Sampler Maintenance, Calibration, and Quality Control

To insure proper operation of automatic samplers, and thus the collection of representative samples, the following maintenance and calibration procedures should be used and any deviations should be documented in the log book.

Prior to being used, the sampler operation will be checked by Field Equipment Center personnel. This includes operation (forward, reverse, automatic) through three cycles of purge-pump-purge; checking desiccant and replacing if necessary; checking the 12-volt batteries to be used with the sampler; and repairing any item if necessary.

During each field trip, prior to initiating the automatic sampler, the rinse and purge-pump-purge cycle shall be checked at least once. The pumping volume should be checked at least twice using a graduated cylinder or other calibrated container prior to initiating the sampler. For flow proportional sampling, the flow pacer that activates the sampler should be checked to insure that it operates properly.

Upon return from a field trip, the structural integrity of the sampler should be examined and repaired, if necessary. The desiccant will be checked and replaced if appropriate. The operation (forward, reverse, automatic, etc.) will be checked and any required repairs will be made and documented. The sampler will then be cleaned as outlined in Appendix B.

The automatic sampler should be checked against the manufacturer's specifications and documented whenever one or more of the sampler functions appears to be operating improperly.

9.5 Manual Sampling

Manual sampling is normally used for collecting grab samples and/or for immediate in-situ field analyses. However, it can also be used in lieu of automatic equipment over extended periods of time for composite sampling, especially when it is necessary to evaluate unusual waste stream conditions.

The best method to manually collect a sample is to use the actual sample container which will be used to transport the sample to the laboratory. This eliminates the possibility of contaminating the sample with intermediate collection containers. If the water or wastewater stream cannot be physically reached by the sampling personnel or it is not safe to reach for the sample, an intermediate collection container may be used, from which the sample can be redistributed to other containers. If this is done, however, the container used to collect the sample must be properly cleaned (Appendix B) and must be made of a material that meets the requirements of the parameter(s) being investigated. Samples for oil and grease, bacteria, phenols, volatile organic compounds, and sulfides analyses must always be collected directly into the sample container.

In some cases it may be best to use a pump, either power or hand operated, to withdraw a sample from the water or wastewater stream. If a pump is used, it is imperative that all components of the pump that come in contact with the sample are properly cleaned (Appendix B) to insure the integrity of the sample.

In general, samples are manually collected by first selecting a location in the wastestream that is well mixed (Section 9.1) then dipping the container in the water or wastewater stream so the mouth of the container faces upstream. The container should not be overfilled if preservatives are present in the container.

9.6 Special Sample Collection Procedures

9.6.1 Organic Compounds and Metals

Trace organic compounds and metals detection limits are usually in the parts per billion or parts per trillion range, so extreme care must be exercised to insure sample integrity.

All containers, composite bottles, tubing, etc., used for sample collection for trace organic compounds and metals analyses should be prepared as described in Appendix B.

When possible, the sample should be collected directly into the appropriate sample container. If the material to be sampled cannot be physically reached, an intermediate collection device may be used. This should be a Teflon®, glass, or stainless steel vessel on a pole or rope or Teflon® tubing via a peristaltic type pump and a Teflon® vacuum container attachment which converts a sample container into a vacuum container. The device which is used should be cleaned as described in Appendix B.

9.6.2 Bacteriological

Samples for bacteriological analyses must always be collected directly into the prepared glass or plastic sample container. The sample container should be kept unopened until it is to be filled. When the cap is removed, care should be taken not to contaminate the cap or the inside of the bottle. The bottle should be held near the base and filled to within about one inch of the top without rinsing and recapped immediately. During sample collection, the sample container should be plunged with the neck partially below the surface and slightly upward. The mouth should be directed against the current. Appendix A lists preservation procedures and holding times.

When the sample container must be lowered into the waste stream, either because of safety or impracticality (manhole, slippery effluent area, etc.), care must be taken to avoid contamination.

9.6.3 Immiscible Liquids/Oil and Grease

Oil and grease may be present in wastewater as a surface film, an emulsion, a solution, or as a combination of these forms. Since it is very difficult to collect a representative sample for oil and grease analysis, the inspector must carefully evaluate the location of the sampling location. The most desirable sampling location is the area of greatest mixing. Quiescent areas should be avoided. The sample container should be plunged into the wastewater using a swooping motion with the mouth facing upstream. Care should be taken to insure that the bottle does not over fill during sample collection.

Because losses of oil and grease will occur on sampling equipment, an automatic sampler should not be used to collect samples for oil and grease analysis. Individual portions collected at prescribed time intervals must be analyzed separately to obtain the average concentrations over an extended period.

9.6.4 Volatile Organic Compounds

Samples to be analyzed for volatile organic compounds (VOCs) should be collected in 40-ml septum vials with screw caps with a Teflon® lined silicone disk in the cap to prevent contamination of the sample by the cap. The disks should be placed in the caps (Teflon® side to be in contact with the sample) in the laboratory prior to the beginning of the sampling program.

When sampling for VOCs, triplicate samples should always be collected from each location. The investigator should determine if the water to be sampled contains chlorine. If the water contains no chlorine, three pre-preserved 40-ml vials should be filled with the sample. The samples may be held for up to 14 days before analysis. When preservation is not feasible, samples can be held up to 7 days before analysis.

If the water contains chlorine, fill an 8-ounce sampling container with 2 drops of a 25% ascorbic acid solution and the water sample. Cap and mix thoroughly but gently by swirling to eliminate residual chlorine. Transfer the sample to three pre-preserved 40-ml vials (see Appendix A). The ascorbic acid and preservative must be added in this order and in two separate steps.

The 40-ml vials should be completely filled to prevent volatilization, and extreme caution should be exercised when filling each vial to prevent any turbulence which could also produce volatilization. The sample should be carefully poured down the side of the vial to minimize turbulence. As a rule, it is best to gently pour the last few drops into the vial so that surface tension holds the water in a "convex meniscus." The cap is then applied and some overflow is lost, but air space in the bottle is eliminated. After capping, turn the bottle over and tap it to check for bubbles; if any are present, repeat the procedure using a new 40-ml vial.

Sampling containers with preservatives should be prelabeled (i.e., P) prior to any field activities. This will reduce the chances of confusion during sampling activities by the investigation team. Sample preservation, containers, holding times, and sample volumes are listed in Appendix A.

9.7 Special Process Control Samples and Tests

During diagnostic evaluations, process control tests may be conducted to evaluate and troubleshoot the performance of the biological treatment processes of a municipal or industrial wastewater treatment facility. The EPA Activated Sludge Process Control Testing handbook is the standard reference for activated sludge process control testing (3). The manual includes a complete description of the step-by-step procedures for each test and the interpretation of the results. The six basic activated sludge process control tests are:

- Sludge settleability (settlometer).
- Centrifuge spins.
- Aeration basin DO profiles.
- Oxygen uptake rate (OUR) measurements.
- Mixed liquor microscopic examinations.
- Sludge blanket depth (SBD) measurements.

Additional references are available that provide a more comprehensive evaluation of the methods used to conduct a diagnostic evaluation (4, 5, 7, 8, and 9). Completion of the Sacramento Operation of Wastewater Treatment Plants course is highly recommended for all personnel prior to serving as the project leader on a Diagnostic Evaluation (6).

9.8 Supplementary Data Collection

While conducting wastewater sampling, the following information will also be obtained (if applicable):

- Field measurements -- pH, dissolved oxygen, conductivity, and temperature (see Section 16 for standard field analytical techniques).
- Flows associated with the samples collected -- continuous flows with composite samples and instantaneous flows with grab samples (Section 18).
- Diagrams and/or written descriptions of the wastewater treatment systems (if available).
- Photographs of pertinent wastewater associated equipment, such as flow measuring devices, treatment units, etc. (keep photolog as specified in section 3.2).
- Process control information on the wastewater treatment process (if applicable).
- Completion of applicable forms required during specific investigations.

All observations, measurements, diagrams, etc., will be entered in bound field logbooks or attached thereto (where applicable as specified in Section 3.5).

9.9 References

1. NPDES Compliance Inspection Manual, United States Environmental Protection Agency, Office of Environment and Compliance Assurance, September 1994.

2. Code of Federal Regulations, 40 CFR, Part 136.3, Table II, (latest issue).

3. US-EPA, "Activated Sludge Process Control Testing", ESD, Water Compliance Unit, Athens, GA, 1990.

4. US-EPA, "Process Control Manual: Aerobic Biological Treatment Facilities MD-14", EPA 430/09-77-006, Office of Water, Washington, D.C., 1977.

5. Metcalf and Eddy, Inc., "Wastewater Engineering: Treatment, Disposal, Reuse", McGraw-Hill Book Co., New York, NY, 1991.

6. California State University - Sacramento, "Operation of Wastewater Treatment Plants - Volumes I, II, III", Sacramento, California.

7. US-EPA, "Retrofitting POTWs", EPA 625/6-89/020, Center for Environmental Research Information, Cincinnati, Ohio, 1989.

8. “Operation Of Municipal Wastewater Treatment Plants”, WEF Manual Of Practice No.11, Water Pollution Control Federation, Alexandria, Virginia, 1990.

9. “Design Of Municipal Wastewater Treatment Plants”, WEF Manual Of Practice No. 8, Book Press, Inc., Brattleboro, Vermont, 1991.